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INDEXED POWDER DIFFRACTION DATA ON DIHYDROXY-FUMARIC ACID, ANHYDROUS, $C_4H_4O_6$

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Chemical evidence that dihydroxyfumaric acid, $C_4H_4O_6$, has the trans-configuration has been given by Hartree (1953) while conclusive evidence regarding this acid having the trans-configuration in the solid state has also been given by Gupta (1953) using single crystals of dihydroxyfumaric acid, dihydrate, $C_4H_4O_6 \cdot 2H_2O$ and X-ray methods. Attempts to crystallize the anhydrous acid (i.e. without the molecules of water of crystallization) from absolute alcohol and other organic solvents by surrounding the solution with a packing of solid carbon dioxide have proved unsuccessful. The powder diffraction data of the anhydrous acid, however, are of such simplicity (figure 1) that it has been possible

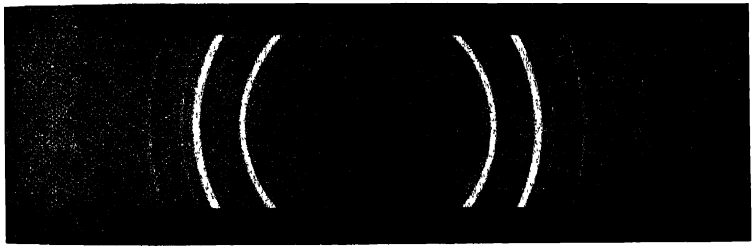

Fig. 1.

to index the data unambiguously after only a few trial combinations. This indexing leads to an orthorhombic cell with

$$a = 12.93\text{Å}, \qquad b = 9.47\text{Å}, \quad c = 8.82\text{Å}.$$

The volume of the cell so chosen is 1081.2A^3. With the measured density of the powder material (1.80 gms/cc) this gives 8 formula units ($C_4H_4O_6$) of the anhydrous acid in the cell. Whether the cell is primitive or non-primitive, the available powder data are insufficient to give any correct indication. However, the very marked simplicity of the powder data and the absence of a large number of lines of simple indices indicate that both structurally as well as from the

space group symmetry the crystal would tend to show a hypercentric distribution (Lipson and Woolfson, 1952).

The powder diffraction data are given in Table I. Corrections for film shrinkage have been applied. The diffraction angles were checked independently by obtaining a diffraction curve on the Norelco Geiger counter diffractometer. There was good agreement regarding the diffraction angles but agreement with the film-method regarding intensity was only qualitative which may be due to preferred orientation in the sample prepared for the diffractometer.

TABLE 1

X-ray diffraction powder data

I/I_1	d (A)		hkl	I/I_1	d (A)		hkl
	Observed	Calculated			Observed	Calculated	
3	6.47	6.47	200	5	2.15	2.16	{014}
						2.15	{241}
1	4.73	4.73	020			2.15	{033}
3	4.41	4 41	002	1	2.04	2.04	{214}
						2 04	{233}
50	4.16	4.17	{102}				
			{021}	1	.98	1.98	{124}
						1.98	{423}
2	3.41	3 40	212				
				7	1.93	1.94	602
100	3.08	3.07	{410}				
		4.07	{130}	1	1.82	1.82	{250}
		3.06	{302}			1.82	{404}
15	2.84	2.84	230	1	1.76	1.75	442
1	2.69	2.70	231	1	1.69	1.69	721
1	2.58	2.57	213	1	1.59	1.59	640
20	2.45	2.45	123	10	1.54	1.54	604
1	2.31	2.33	{140}				
		2.33	{223}				

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